CONTRACT REPORT NO. OSQ84-00174

RECOMMENDED VALUES FOR REFERENCE ALLOYS NZA-1 to NZA-7:

RESULTS OF SAMPLE ANALYSIS

23440-4-9155-4

PREPARED FOR

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JUL 03 1985

MICROMEDIA

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11 Ag

March 1985

1. Introduction

Reliable reference standards are essential for establishing quality control in analytical laboratories. Although rock and soil standards are readily available from various government agencies, it is often difficult to obtain suitable standards for the analysis of certain specific materials, such as alloys.

This report describes the results of the analysis of seven Zn/Al alloys for Al, Cd, Cu, Fe, Mg, Pb and Sn, with the objective of establishing recommended values for reference alloys to be used as statistical means of controlling precision and accuracy in analytical laboratories.

2. Analytical Methodology

The following analytical techniques were used to determine Al, Cd, Cu, Fe, Mg, Pb, and Sn. Each of the samples submitted was analysed five times.

2.1 Determination of Al:

A 0.20 - 1.00 gm. aliquot of sample was dissolved in conc. HCl with sufficient hydrogren peroxide to complete dissolution. Aluminum is then precipitated from this solution in an NH_4Cl medium with NH_4OH to a methyl red end point. After filtering and redissolving in HCl, the aluminum hydroxide is complexed with EDTA, and the pH adjusted to 5.5. The excess EDTA is titrated with zinc to a xylenol orange end point.

Proof aluminum is carried through as standards and per cent aluminum is calculated from the obtained titre.

2.2 Determination of Cd, Cu, Fe, Mg and Pb

A 1.000 gram aliquot of each samples was digested in conc. HCl and refluxed for 2 hours in beakers covered with watch glasses. The samples were then evaporated to dryness. The residue was dissolved in 6M HCl and the solution diluted to 50 ml. in a volumetric flask.

Concentrations of Cd, Cu, Fe, Mg and Pb were determined by direct aspiration of the solution into a calibrated IL-457 atomic absorption spectrophotometer.

2.3 Determination of Sn

An aliquot of the solution prepared in 2.2 above is removed. The tin is converted to tin iodide and extracted into toluene. After back-extracting into a nitric-hydrochloric-sulfuric acid solution, the tin is determined by atomic absorption spectrophotometry.

Detection levels for all techniques used to determine elements in the seven alloy samples are given in Table 1.

3. Results and Discussion

Results of analyses of Zn/Al alloys NZA-1 to NZA-7 are given in Table 2. The mean values for five determinations of each element with relative standard deviations are tabulated in Table 3.

With the exception of all Sn values, Mg in sample NZA-3 and NZA-4, and Pb in NZA-5, all % RSD values are below 5%. The poor precision for Sn may reflect the low element concentration in the samples due to instrumental detection limit for the procedure chosen.

Table 1

Detection Limits for Elements Determined in Samples NZA 1-7.

Element	Detection Limit
Al	0.1 %
Cd	0.5 ppm
Cu	0.01 %
Fe	5 ppm
Mg	5 ppm
Pb	1 ppm
Sn	10 ppm

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PROJECT: 02SQ.23440-4

WORK ORDER: 8045D-85
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GEOCHEMICAL LABORATORY REPORT

SAMPLE TYPE: ALLOY

SAMPLE NUMBER		AL %	CD PPM	CU %	FE PPM
NZA-1:	1	28.3	8.1	1.59	514.0
NZA-1:	2	28.6	8.3	1.43	517.0
NZA-1:	3	28.0	8.0	1.5	511.0
NZA-1:	4	28.1	8.3	1.43	513.0
NZA-1:	5	28.3	8.3	1.44	515.0
NZA-2:	1	24.2	39.0	2.83	240.0
NZA-2:	2	24.3	43.0	2.93	246.0
NZA-2:	3	23.9	44.0	2.86	239.0
NZA-2:	4	23.7	42.0	2.75	243.0
NZA-2:	5	23.9	42.0	2.86	237.0
NZA-3:	1	26.4	40.0	1.95	730.0
NZA-3:	2	26.2	43.0	1.99	740.0
NZA-3:	3	26.0	41.0	2.01	750.0
NZA-3:	4	26.0	41.0	1.99	730.0
NZA-3:	5	26.3	40.0	1.96	750.0
NZA-4:	1	26.9	26.0	1.85	295.0
NZA-4:	2	27.1	24.0	1.86	299.0
NZA-4:	3	26.8	27.0	1.93	295.0
NZA-4:	4	26.6	26.0	1.91	296.0
NZA-4:	5	26.9	25.0	1.85	298.0
NZA-5:	1	10.9	84.0	1.12	135.0
NZA-5:	2	10.7	84.0	1.08	141.0
NZA-5:	3	11.0	84.0	1.16	136.0
NZA-5:	4	10.7	86.0	1.13	140.0
NZA-5:	5	10.8	85.0	1.14	138.0
NZA-6:	1	7.46	130.0	2.69	117.0
NZA-6:	2	7.29	131.0	2.7	115.0
NZA-6:	3	7.59	130.0	2.68	115.0
NZA-6:	4	7.39	132.0	2.73	118.0
NZA-6:	5	7.45	129.0	2.86	116.0

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GEOCHEMICAL LABORATORY REPORT

SAMPLE TYPE: ALLOY

SAMPLE NUMBER		AL %	CD PPM	CU %	FE PPM
NZA-7:	1	13.3	<0.5	0.2	137.0
NZA-7:	2	13.1	<0.5	0.2	138.0
NZA-7:	3	13.4	<0.5	0.2	143.0
NZA-7:	4	12.9	<0.5	0.21	139.0
NZA-7:	5	13.3	<0.5	0.2	140.0

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GEOCHEMICAL LABORATORY REPORT

SAMPLE TYPE: ALLOY

SAMPLE NUMBER		MG PPM	PB PPM	SN PPM
NZA-1:	1	185.0	38.0	80.0
NZA-1:	2	165.0	35.0	80.0
NZA-1:	3	170.0	34.0	70.0
NZA-1:	4	175.0	36.0	70.0
NZA-1:	5	170.0	38.0	70.0
NZA-2:	1	255.0	87.0	40.0
NZA-2:	2	260.0	85.0	40.0
NZA-2:	3	275.0	85.0	40.0
NZA-2:	4	255.0	86.0	50.0
NZA-2:	5	275.0	82.0	40.0
NZA-3:	1	50.0	46.0	40.0
NZA-3:	2	60.0	45.0	30.0
NZA-3:	3	55.0	49.0	30.0
NZA-3:	4	50.0	45.0	40.0
NZA-3:	5	55.0	46.0	40.0
NZA-4:	1	105.0	105.0	100.0
NZA-4:	2	115.0	99.0	80.0
NZA-4:	3	115.0	95.0	80.0
NZA-4:	4	95.0	94.0	90.0
NZA-4:	5	110.0	96.0	90.0
NZA-5:	1	195.0	14.0	30.0
NZA-5:	2	200.0	15.0	50.0
NZA-5:	3	200.0	13.0	40.0
NZA-5:	4	195.0	19.0	40.0
NZA-5:	5	205.0	15.0	40.0
NZA-6:	1	<5.0	710.0	120.0
NZA-6:	2	<5.0	700.0	100.0
NZA-6:	3	<5.0	690.0	90.0
NZA-6:	4	<5.0	720.0	90.0
NZA-6:	5	<5.0	720.0	100.0

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GEOCHEMICAL LABORATORY REPORT

SAMPLE TYPE: ALLOY

SAMPLE NUMBER		MG PPM	PB PPM	SN PPM
NZA-7:	1	550.0	132.0	200.0
NZA-7:	2	560.0	136.0	180.0
NZA-7:	3	570.0	134.0	170.0
NZA-7:	4	560.0	134.0	160.0
NZA-7:	5	545.0	133.0	160.0

SIGNED: C. Read

C. Douglas Read.
LABORATORY MANAGER

FOOTNOTES:
P=QUESTIONABLE PRECISION; *=INTERFERENCE; TR=TRACE; ND=NOT DETECTED;
IS=INSUFFICIENT SAMPLE; NA=NOT ANALYZED; MS=MISSING SAMPLE

Table 3

Mean & Percent Relative Standard Deviation
Values for Elements Determined in Samples NZA 1-7

STANDARD:		NZA-1	NZA-2	NZA-3	NZA-4	NZA-5	NZA-6	NZA-7
Al	Mean %	28.3	24.0	26.2	26.9	10.8	7.44	13.2
	RSD %	.83	1.02	.68	.69	1.22	1.47	1.52
Cd	Mean ppm	8.2	42.0	41.0	25.6	85.0	130	<.5
	RSD %	1.72	4.45	2.99	4.45	1.18	.94	0
Cu	Mean %	1.48	2.85	1.98	1.88	1.13	2.73	.20
	RSD %	4.66	2.29	1.23	1.99	2.65	2.71	2.50
Fe	Mean ppm	514	241	740	297	138	116	139
	RSD %	.43	1.46	1.35	.63	1.85	.96	1.69
Mg	Mean ppm	169	264	54	108	199	<5	557
	RSD %	2.47	3.88	7.75	7.74	2.10	0	1.75
Pb	Mean ppm	36	85	46	98	15	708	134
	RSD%	5.00	2.20	3.60	4.53	15.3	1.84	1.11
Sn	Mean ppm	74	42	36	88	40	100	174
	RSD%	7.4	10.6	15.2	9.5	17.6	12.2	8.1

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